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13. ABSTRACT (Maximum 200 words) The objective of the program is to elucidate novel concepts (including micro-structures, processes, and materials) in the following 2 key areas of ceramics applications relevant to the US Air Force: (i) contact-damage resistance and (ii) thermal insulation/protection. During the period covered by this report, major innovations in the above 2 areas have been achieved and are summarized.				
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FINAL AFOSR GRANT REPORT

Fundamental Studies of Novel Contact-Damage Resistant Ceramics

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1. Objective

The objective of the program was to elucidate novel concepts (including microstructures, processes, and materials) in the following 2 key areas of ceramics applications relevant to the Air Force: (i) contact-damage resistance and (ii) thermal insulation/protection.

2. Status of Effort

During this three year grant major innovations in the above 2 areas were achieved and are given below [1-5].

3. Accomplishments/New Findings

3.1. Contact Damage Resistant Ceramics

Using elastic Hertzian (spherical) indentation experiments in a graded alumina-glass composite we have demonstrated that cone-cracking in this composite can be completely suppressed, solely by the virtue of the gradation of its elastic modulus, E [1,2]. Here E increases with the depth (z) below the indented surface. Under the same indentation conditions fully-developed cone cracks are produced in both the bulk alumina and the bulk glass. A similar beneficial, crack-suppression effect was observed in sliding contact as well [3].

A novel, *in situ* processing method was employed to fabricate easily the above graded alumina-glass composite, free of macroscopic residual stresses. This method involves the impregnation of a dense, fine-grained alumina by a low-elastic-modulus, aluminosilicate glass at elevated temperatures, where the glass penetrates the alumina grain boundaries.

Analytical and finite-element models describing Hertzian indentation of elastically-graded materials [6-8], which were used to guide the present experiments, have shown that the suppression of Hertzian cracks in the graded alumina-glass composite is due to the diffusion of the principal tensile stresses into the interior.

This method was extended to the system of silicon nitride-oxynitride glass system, and the same effect of crack suppression was demonstrated. There is a perceived disadvantage to having large amounts of glass at the contact surface with regards to wear properties. Therefore, a new system of silicon nitride-silicon carbide was invented, where there is silicon nitride at the surface grading down to silicon carbide in the interior. We have demonstrated that such graded ceramics can be processed and sintered without using external pressure. These new graded ceramics show the same quality of crack suppression.

This novel approach of elastic-modulus grading for the enhancement of contact-damage resistance in otherwise brittle materials has a generic appeal, with potential applicability in structural parts, microelectronics (devices and manufacturing), dental materials, prosthesis, magnetic materials, concrete, etc. Furthermore, since the contact-damage resistance effect in graded materials is purely elastic, it is suggested that this effect is likely to be inherently immune to mechanical fatigue. Also, since grain boundaries in most ceramics can be penetrated by glasses of appropriate compositions [9], the novel, *in situ* processing method described above is potentially applicable to a broad range of ceramics such as oxides, mixed oxides, nitrides, oxynitrides, carbides, silicides, etc.

3.2. Thermal-Barrier Ceramics

With regards to thermal-barrier ceramics, an alternative family of ceramics — garnets — has been identified to replace the currently used yttria-stabilized zirconia (YSZ) ceramics in thermal barrier coatings (TBCs) for potentially improved durability [4,5]. TBCs used to insulate and protect hot-section metal components in gas-turbine engines fail prematurely by spallation during service, exposing the bare metal to dangerously hot gases [9]. This issue of TBC failure becomes increasingly important as future high-efficiency gas-turbine engines are designed to operate at increasingly higher temperatures. Of the various factors contributing to TBC failures, one of the most important factor is the oxidation of the metal bond coat [10], a thin bonding layer (10 to 50 μm thickness) sandwiched between the metal substrate and the YSZ coating (125 to 500 μm thickness). The engine environment is the source for the oxygen, which is readily transported through the YSZ coating to the bond coat. The oxygen transport occurs by fast diffusion if the microscopic defects (microcracks, pores), that are usually present in the zirconia TBCs, form a non-percolating network. The very high concentrations of oxygen vacancies present in YSZ, which contribute to the high thermal resistivity in YSZ, are also responsible for the fast diffusion of the oxygen — a factor that appears to fundamentally limit the useful life of YSZ-based TBCs.

Of the various approaches proposed to address the bond-coat oxidation issue, the notion of replacing YSZ with an alternative ceramic of high thermal resistivity, but low oxygen diffusivity, has perhaps the most far-reaching implications, and forms the basis for the present study. In the course of a systematic search, we have found that yttrium aluminum garnet or YAG ($\text{Y}_3\text{Al}_5\text{O}_{12}$) may offer a possible alternative to traditional YSZ [4, 5].

YAG is being considered for future high-temperature structural applications [11] for its excellent high-temperature mechanical properties [11] and phase/thermal stability. Moreover, oxygen diffusivity in dense, polycrystalline YAG ($\approx 10^{-20} \text{ m}^2\text{s}^{-1}$ at 1100 $^\circ\text{C}$ [12]) is ≈ 10 orders of magnitude lower than that in YSZ ($\approx 10^{-10} \text{ m}^2\text{s}^{-1}$ @ 1100 $^\circ\text{C}$ for YSZ). Also YAG has a high thermal expansion coefficient (low thermal-expansion mismatch with metals). All these attributes make YAG a potential candidate for alternative TBC ceramic. However, high-temperature thermal conductivity of polycrystalline YAG had not been documented in the open literature.

We have measured the thermal conductivity of dense, polycrystalline YAG as a function of temperature and have found that the high-temperature thermal conductivity of YAG is comparable to that of YSZ [4,5]. Furthermore, it is possible to tailor the thermal resistivity of YAG by alloying it with other ceramics from the isostructural family of garnets — a generic approach [13], potentially applicable to other garnets besides YAG. This concept is illustrated here in the case of YAG-YIG solid solutions, where YIG is yttrium iron garnet ($\text{Y}_3\text{Fe}_5\text{O}_{12}$). The effects of temperature and composition on the observed thermal conductivities have been interpreted with reference to established theories for thermal conduction by phonons and photons [13]. Currently, microstructures of garnets are being tailored to achieve even lower thermal conductivities. For example, by reducing the grain size into the nm region one can enhance the phonon scattering. Simultaneously, by introducing controlled μm -size porosity one can scatter photons [13].

With regards to processing, there is no fundamental barrier to the processing of garnet-based TBCs either by plasma spraying or by electron beam physical vapor deposition (EB-PVD), the two most important methods of TBC manufacturing.

3.3. References

1. J. Jitcharoen, N.P. Padture, A.E. Giannakopoulos and S. Suresh, Hertzian-Crack Suppression in Ceramics with Elastic-Modulus-Graded Surfaces, 1998, Journal of the American Ceramic Society, 81: 2301-308.
2. S. Suresh, A.E. Giannakopoulos, N.P. Padture and J. Jitcharoen, Method and Apparatus for Determination of Mechanical Properties of Functionally-Graded Materials, 1997, U.S. Patent (filed).
3. S. Suresh, M. Olsson, A.E. Giannakopoulos, N.P. Padture and J. Jitcharoen, Engineering the Resistance to Sliding-Contact Damage Through Controlled Gradients in Elastic Properties at Contact Surfaces, 1999, Acta Mater. in press.
4. N.P. Padture and P.G. Klemens, Low Thermal Conductivity in Garnets, 1997, Journal of the American Ceramic Society, 80: 1018-1020.
5. N.P. Padture, M. Gell and P.G. Klemens, Ceramic Materials for Thermal Barrier Coatings and Method of Identification, 1997, U.S. Patent, (filed).
6. S. Suresh, A.E. Giannakopoulos and J. Alcala, Spherical Indentation of Compositionally Graded Materials: Theory and Experiments, 1997, Acta Materialia, 45: 1307-1321.
7. A.E. Giannakopoulos and S. Suresh, Indentation of Solids with Gradient in Elastic Properties: Part I, Point Force, 1997, International Journal of Solids and Structures, 34: 2357-2392.
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9. S.M. Meier, D.K. Gupta and K.D. Sheffler, Ceramic Thermal Barrier Coatings for Commercial Gas Turbine Engines, 1991, Journal of Metals, 43 :50-53.
10. R.A. Miller and C.E. Lowell, Failure Mechanisms of Thermal Barrier Coatings Exposed to Elevated Temperatures, 1982, Thin Solid Films, 95: 265-273.
11. T.A. Parthasarthy, T.-I. Mah and K. Keller, Creep Mechanism of Polycrystalline Yttrium Aluminum Garnet, 1992, Journal of the American Ceramic Society, 75: 1756-1759.
12. I. Sakaguchi, H. Haneda, J. Tanaka and T. Yanagitani, Effect of Composition on the Oxygen Tracer Diffusion in Transparent Yttrium Aluminum Garnet (YAG) Ceramics, 1996, Journal of the American Ceramic Society, 79: 1627-1632.
13. P.G. Klemens, Theory of the Thermal Conductivity of Solids, 1969, in Thermal Conductivity 1, Academic Press, London, UK: 1-68.
14. P.G. Klemens, Theory of Thermal Conductivity of Nanophase Materials, 1997, in Chemistry and Physics of Nanostructures and Related Non-Equilibrium Materials, TMS, Warrendale, PA, USA: 97-104.

4. Personnel

4.1. Personnel Supported

<u>Faculty:</u>	Prof. Nitin P. Padture, PI
<u>Post-Doc:</u>	Dr. Vijay V. Pujar, 50% effort
<u>Graduate Students:</u>	Ms. Juthamas Jitcharoen, 100% effort Mr. David C. Pender, 100% effort

4.2. Personnel Associated

Faculty:
Prof. Paul G. Klemens, UConn
Prof. Maurice Gell, UConn
Prof. Subra Suresh, MIT
Prof. Antonios E. Giannakopoulos, MIT

5. Resulting Theses

1. Juthamas Jitcharoen, 1999, Ph.D. "Contact-Damage Resistance in Alumina-Based Ceramics with Elastic-Modulus-Graded Surfaces" University of Connecticut. Now on the faculty of Ubon Ratachathani University, Thailand.
2. David C. Pender, 1999, Ph.D. "Ceramics with Graded Surfaces for Contact Damage Resistance" University of Connecticut. Now at General Electric Corporate R&D, Schenectady, NY.

6. Publications

1. J. Jitcharoen, N.P. Padture, A.E. Giannakopoulos and S. Suresh, Hertzian-Crack Suppression in Ceramics with Elastic-Modulus-Graded Surfaces, 1998, Journal of the American Ceramic Society, 81: 2301-308.
2. S. Suresh, M. Olsson, A.E. Giannakopoulos, N.P. Padture and J. Jitcharoen, Engineering the Resistance to Sliding-Contact Damage Through Controlled Gradients in Elastic Properties at Contact Surfaces, 1999, Acta Mater. in press.
3. N.P. Padture and P.G. Klemens, Low Thermal Conductivity in Garnets, 1997, Journal of the American Ceramic Society, 80: 1018-1020.
4. N.P. Padture, D.C. Pender and J. Jitcharoen, Microstructural Tailoring of Ceramics for Mechanical Properties, 1998, Proceedings of the VIth National Congress on the Mechanical Properties of Solids, Eds. A. Pajares, F.L. Cumbreira and F. Guiberteau, University of Extremadura, Badajoz, Spain: 19-29.

7. Presentations

7.1. Invited Presentations

1. N.P. Padture, "Microstructural Tailoring of Ceramics for Mechanical and Thermal Properties," Meeting of the Connecticut Chapter of the TMS, Cromwell, CT, March, 1997.
2. N.P. Padture, "Novel Concepts in Contact-Damage-Resistant Ceramics and Thermal-Barrier Ceramics," National Institute of Standards and Technology, Gaithersburg, MD, March, 1997.
3. N.P. Padture, "Microstructural Tailoring of Ceramics for Mechanical Properties," Yale University, New Haven, CT, April, 1997.
4. N.P. Padture, "Microstructural Tailoring of Ceramics for Mechanical Properties" University of Connecticut Physics Department, Storrs, CT, April, 1997.
5. N.P. Padture, "Novel Concepts in Contact-Damage-Resistant Ceramics and Thermal-Barrier Ceramics," AFOSR Annual Review Meeting, Cincinnati, OH, May, 1997.
6. N.P. Padture, "Microstructural Tailoring of Structural Ceramics: Challenges, Opportunities, and Basic Research Needs," NSF Workshop on the Fundamental Research Needs in Ceramics, Arlington, VA, June, 1997.

7. "Microstructural Tailoring of Ceramics for Mechanical and Thermal Properties"
N.P. Padture
International Materials Research Congress, Cancún, Mexico, September 1997
8. "Tailoring of Ceramic Micro-and Macro-Structures for Mechanical Properties"
N.P. Padture
Purdue University, West Lafayette, IN, November 1997
9. "Novel Concepts in Contact-Damage-Resistant Ceramics and Thermal-Barrier Ceramics"
N.P. Padture
Meeting of the New England Chapter of the American Ceramic Society, Auburn, MA, December 1997
10. "Microstructural Tailoring of Ceramics for Mechanical Properties"
N.P. Padture
Olin Corporation, New Haven, CT, February 1998
11. "Microstructural Tailoring of Ceramics for Mechanical and Thermal Properties"
N.P. Padture
State University of New York, Stony Brook, NY, April 1998
12. "NSF Workshop on Fundamental Research Needs in Ceramics: Report of the Working Group on Structural and Electromechanical Ceramics"
N.P. Padture
100th Annual Meeting of the American Ceramic Society, Cincinnati, OH, May 1998
13. "YAG-Based Thermal Barrier Coatings"
N.P. Padture
US-European Joint Workshop on Thermal Barrier Coatings, Irsee, Germany, May 1998
14. "Microstructural Tailoring of Ceramics for Mechanical Properties"
N.P. Padture
VIth Conference on the Mechanical Properties, Badajoz, Spain, June 1998
15. "*In Situ* Processing of and Hertzian-Crack Suppression in Novel Ceramics Microstructures"
N.P. Padture, D.C. Pender and J. Jitcharoen
9th World Ceramics Congress (CIMTEC '98), Florence, Italy, June 1998

7.2. Contributed Presentations

1. N.P. Padture, D.C. Pender, H. Ye and V.V. Pujar, "*In Situ* Processing and Mechanical Behavior of Monolithic and Layered Ceramic Composites," Engineering Ceramics Division of the American Ceramic Society, Cocoa Beach, FL, January, 1997.
2. N.P. Padture, P.G. Klemens and M. Gell, "Low Thermal Conductivity in Garnet Ceramics, and their Possible New Use as Advanced Thermal Barrier Coatings," 98th Annual Meeting of the American Ceramic Society, Cincinnati, OH, May, 1997.
3. N.P. Padture, P.G. Klemens and M. Gell, "Low Thermal Conductivity in Garnet Ceramics, and their Possible New Use as Advanced Thermal Barrier Coatings," Interagency Thermal Barrier Coatings Workshop, Cincinnati, OH, May, 1997.
4. "Hertzian-Crack Suppression in Ceramics with Elastic-Modulus Graded Surfaces"
J. Jitcharoen*, N.P. Padture, A.E. Giannakopoulos and S. Suresh
Fall Meeting of the Materials Research Society, December, 1997
5. "Yttrium Aluminum Garnet (YAG) Thermal Barrier Coatings for Improved Durability"
N.P. Padture*, V.V. Pujar and S. Sampath
100th Annual Meeting of the American Ceramic Society, Cincinnati, OH, May, 1998

6. "Hertzian-Crack Suppression in Ceramics with Elastic-Modulus Graded Surfaces"
J. Jitcharoen*, N.P. Padture, A.E. Giannakopoulos and S. Suresh
100th Annual Meeting of the American Ceramic Society, Cincinnati, OH, May, 1998
7. "Silicon Nitride-Based Ceramics with Elastic Modulus-Graded Surfaces for Contact-Damage Resistance"
D.C. Pender* and N.P. Padture
100th Annual Meeting of the American Ceramic Society, Cincinnati, OH, May, 1998

* Presenting author

8. Patent Disclosures

1. S. Suresh, A.E. Giannakopoulos, N.P. Padture and J. Jitcharoen, "Method and Apparatus for Determination of Mechanical Properties of Functionally-Graded Materials," U.S. Patent, filed (1997).
2. N.P. Padture, M. Gell and P.G. Klemens, "Ceramic Materials for Thermal Barrier Coatings and Method of Identification," U.S. Patent, filed (1997).

9. Honors/Awards

1. Third Prize for Regular Paper Presented: N.P. Padture, D.C. Pender, H. Ye and V.V. Pujar, "*In Situ* Processing and Mechanical Behavior of Monolithic and Layered Ceramic Composites," Engineering Ceramics Division of the American Ceramic Society, Cocoa Beach, FL, January, 1997.
2. Third Prize for Ceramographic Poster in Combined Techniques Category: J. Jitcharoen and N.P. Padture, "Hertzian-Cone-Crack Suppression in Functionally-Graded Ceramics," 99th Annual Meeting of American Ceramic Society, Cincinnati, OH, May, 1997.
3. N.P. Padture received the Olin Junior Faculty Development Award from Olin Corporation, New Haven, CT, February, 1998.
4. N.P. Padture received early promotion to the rank of Associate Professor, 1998 and tenure in 1999.
5. J. Jitcharoen Received Third Prize for student poster: "Hertzian-Crack Suppression in Ceramics with Elastic-Modulus Graded Surfaces" at the New England Meeting of American Ceramic Society, Waltham, MA, June, 1998.
6. N.P. Padture received the Outstanding Junior Faculty Award, for "outstanding scholarly achievements and sustained future professional growth" from the University of Connecticut School of Engineering, 1998.
7. N.P. Padture received the Robert L. Coble Award for Young Scholars, for "outstanding contribution to the understanding and education of the mechanical behavior of ceramics/composites" from the American Ceramic Society, 1999.